

DESIGN AND TESTING OF AN EXPERIMENT TO MEASURE SELF-FILTRATION IN
PARTICULATE SUSPENSIONS

by

Mattias S. Flander

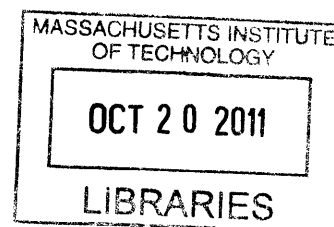
A thesis submitted in partial fulfillment of the requirements for the degree of

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Mattias S. Flander

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ABSTRACT

An experiment for measuring self-filtration in terms of change in volume fraction downstream of a constriction compared to volume fraction upstream of said constriction was designed and tested. The user has the ability to control a variety of parameters including constriction geometry, flow rate, and initial volume fraction in order to evaluate their impact on downstream volume fraction. The relative uncertainty in measured downstream volume fraction was found to be 1.31%. Experimental data was collected to show the effect of changing initial volume fraction and flow rate on downstream volume fraction.

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LIST OF SYMBOLS USED

Symbol	Quantity ^a	Units
Φ_x	Downtown volume fraction	[ml/ml]
Φ_0	Initial upstream volume fraction	[ml/ml]
Φ_m	Particle packing density	[ml/ml]
L	Constriction length	[in]
l	Submersion depth	[in]
D	Constriction Diameter	[in]
d	Particle diameter	[mm]
ρ_f	Fluid density	[g/ml]
ρ_p	Polystyrene density	[g/ml]
v	Fluid velocity	[m/s]
V	Volume	[ml]
μ	Dynamic Viscosity	[Pa-s]
m	mass	[g]
Stk	Stokes Number	[-]
Re	Reynolds Number	[-]

GLOSSARY

Volume Fraction. The solid volume of suspended particles (internal phase) divided by the volume of fluid (external phase) before mixing.

Self-Filtration. A phenomenon where volume fraction downstream of a mechanical constriction is reduced compared to the upstream volume fraction.

Packing Density. The volume fraction of a sediment column.

INTRODUCTION

Quantifying flows of particulate dispersions is important to many applications in industry as well as within the scientific community, yet relatively few publications currently exist on the subject, and it is still not well understood. One such effect is the jamming and unjamming effects when a particulate suspension flows through a constriction, such as a sudden reduction in diameter of a pipe. Another common geometry is the submersion of a suction tube or pipe into a particulate suspension in order to pump it from one container to another. Examples include pumping mud out of a newly dug well, pumping stomach contents from patients with gastro-intestinal problems, as well as the annoying tendency of a “slushy” soft drink to leave all the crushed ice behind when one is trying to drink it with a straw.

This thesis describes the design and testing of an experiment to measure self-filtration by measuring volume fraction downstream of a mechanical constriction. The experimental design draws on aspects from a variety of previously published papers (described in section 1.1) and attempts to improve on those designs. The repeatability of the experiment is analyzed in depth and data is collected and compared to previously published data.

1.1 PREVIOUS WORK

The most important inspiration for designing this experiment came from a paper published by M.D Haw [1] in 2004. In brief, Haw found that volume fraction of hard-sphere colloidal suspensions downstream of a mechanical constriction was reduced by an effect he called “self-filtration”. Through microscopic observation of the flow near the constriction, he observed that the particles went through cycles of “jamming” and “un-jamming” (a conversion from fluid to solid and vice versa) when converging at the constriction inlet. He proposed that during the time that the particles jammed to form a solid, the liquid phase continued to flow through the pores of the temporarily formed solid, thus reducing downstream volume fraction compared to upstream volume fraction. These experiments were conducted with particles in the colloidal region, where Brownian motion dominates, except for one batch where the particle diameter was 1000 ± 50 nm, which is the beginning of the granular region (where gravity is dominant). Haw reported that trials with higher initial volume fraction caused the jammed solid to exist for longer intervals before collapsing compared to trials with lower initial volume fraction. He also reported that below some limiting initial volume fraction, the jammed solid essentially collapsed immediately every time it formed, which resulted in no self-filtration taking place at all.

A more recent publication [2], looked at significantly larger particles, ranging from 0.380-0.500 mm in diameter. While Haw used a 3D convergent flow (a small diameter syringe submerged from above into a beaker full of suspension and the plunger pulled out to create a negative pressure gradient), Kulkarni et al. [2] used an approximation of 1D Poiseuille flow by pouring the suspension into a vertical rectangular channel made from acrylic tubing with an inserted orifice plate as the mechanical constriction. The flow is gravity driven for most trials, but adding weighed pistons on top of the fluid column also increased the upstream pressure for some trials. Volume fraction was measured by thorough washing and drying before weighing the solid, which is similar to the technique employed in [1].

Both Haw and Kulkarni picked convenient methods for driving their flows, which required no expensive precision pumps or flow regulators. However, Haw has no ability to maintain a constant pressure gradient throughout the experiment, and while the gravity driven flow in [2] likely reaches terminal velocity quickly, and the experimenters could increase the driving pressure by adding weighed pistons, the flow rate is still poorly controlled. A publication that focused more on the effects of the driving pressure [3] drove flows of suspension through a constriction by actuating the plunger of a piston with a motor and observing the velocity profiles of the suspension flowing through a constriction using confocal microscopy. One of the main findings of Isa et al. [3] was that the applied pressure gradient played an important role in determining the flow behavior of a suspension at a given particle size and volume fraction. In particular, applied pressures above a certain threshold was found to slow down the flow rather than the speeding up that is observed for Newtonian fluids.

1.2 OBJECTIVE AND FUNCTIONAL REQUIREMENTS

This thesis was conceived as a way of both trying to replicate Haw's findings using suspensions of larger, non-colloidal particles, as well as a way to control and vary certain parameters that previous experiments were unable to control or vary. In addition, this was to be accomplished with a budget on the order of \$100. Only basic laboratory equipment such as scales, specimen vials, pipettes, etc. was available, as well as a DSLR camera. The available solid particles for mixing the suspension were polystyrene beads of in various sizes ranging from 0.15-1.68 mm in diameter. The fluid phase was mixed by hand using tap water, salt and Tween 20 surfactant.

With these goals and restrictions in mind, we decided on a set of "functional requirements" for the experimental design:

1. Measure volume fraction of polystyrene in the suspension upstream and downstream of a constriction.
2. Ability to control as many parameters as possible of the flow path geometry as well as the flow itself.
3. Repeatability of measurements
4. Simple flow geometry
5. Inexpensive and low-tech experiment

One of the parameters that Haw and Kulkarni were unable to control well was the flow rate through the constriction, which could not be maintained at a constant value in [1] due to the fact that the pressure gradient driving the flow decreased in magnitude over the course of extraction. The flow would therefore start out relatively fast and then decelerate until the pressure head of the collected column of suspension balanced the pressure inside the syringe and the suspension stopped flowing altogether. Considering the conclusions of [3] that driving pressure is a fundamentally important quantity for the characterization of these flows, it was desirable to construct a method of driving the flow that allowed precision control of the applied driving pressure, and consequently the flow rate.

Another parameter that neither Haw nor Kulkarni vary is the diameter of the constriction. The existence of a limiting initial volume fraction under which no self-filtration occurs raises the question of which factors determine what this limiting value is. The design of our experiment contains both these modifications along with an alternative way of measuring volume fraction without the use of expensive equipment such as centrifuges that may not be available in some labs. A third desirable modification is the use of simpler flow geometry. Haw's experiment explored a 3D convergent flow from a large volume of surrounding suspension into a submerged syringe. One of the early strategies developed for our experimental design was to have a fluid flow through a pipe into the constriction, which would give an approximately 1D-flow and simplify analysis. The experiment was therefore designed in a way that allows for flows in both the 3D-geometry of [1] as well as a 1D-flow through a pipe before the suspension enters the constriction, as was employed in Kulkarni.

The two strategies that were pursued in the design of the hardware for this experiment were therefore:

1. Driving the suspension through two sections of tubing connected by a small diameter constriction section using a pressure gradient. This could be approximated as a one-dimensional flow.

2. Driving the suspension through a small diameter constriction submerged into a beaker full of suspension using a pressure gradient. This would be a 3D convergent flow.

Due to the limited scope of this thesis, the focus was on replicating Haw's experiment and all data collected and reported here used the 3D convergent geometry. A prototype for a flow tube of the 1D geometry was nonetheless constructed and tested as a proof of concept.

1.3 HARDWARE DESIGN

The pump consists of pneumatic cylinder whose piston is threaded into a block of aluminum that is free to slide on steel guiding rods along the same axis as the piston. This block is in turn coupled to a lead screw that is driven by a bipolar stepper motor capable of 0.8 degree steps. This allows very slow continuous motion of the piston resulting in Reynolds numbers on the order of 1-100 for pure water. The stepper motor also allows very accurate volume sampling, with the largest observed error being about 0.1 ml, presumably due to backlash in the lead screw assembly or an incomplete seal in the flow tube. The stepper motor is controlled using Pulse Width Modulation (PWM) by programming a Basic Stamp microcontroller to feed a square wave to a stepper driver board that was purchased from Sparkfun.

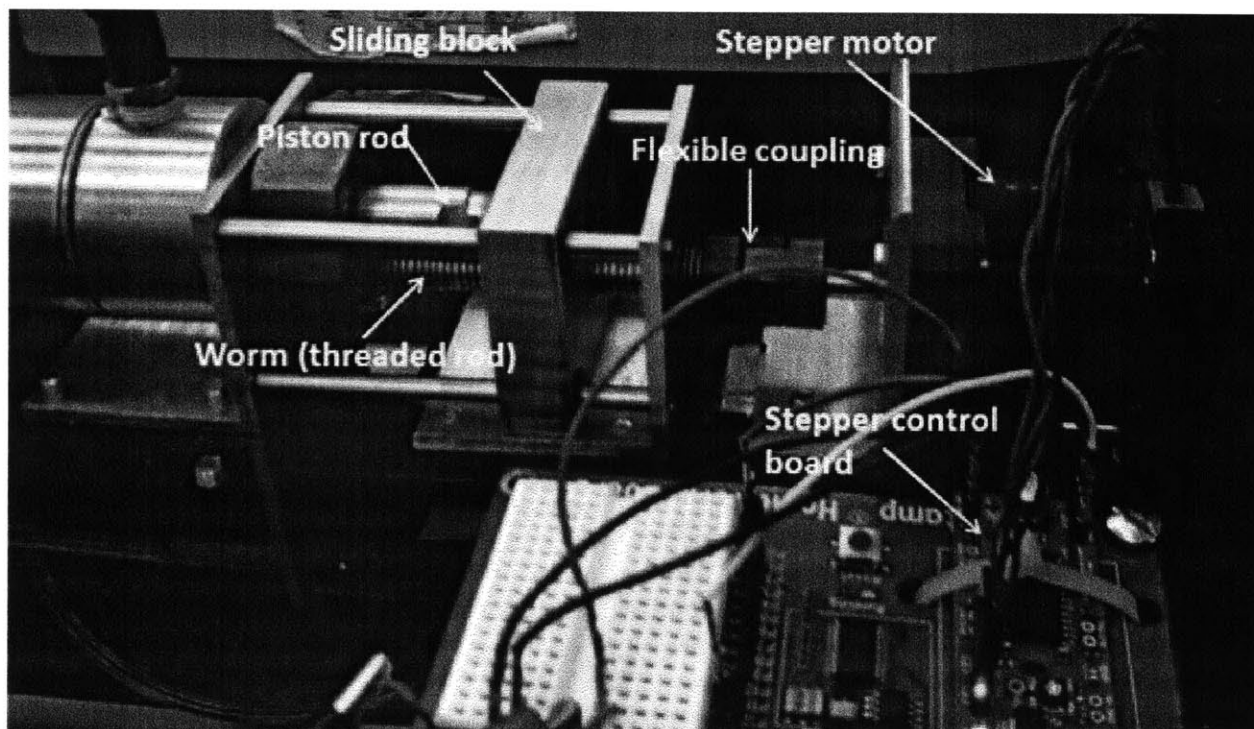


Fig. 1 *Annotated syringe pump assembly*

The first prototype for the pump did not have a lead screw assembly or a stepper motor. Instead, a hacked servomotor was used to pull the piston shaft along with a rack and pinion that were laser cut from acrylic. This first prototype was cheap and easy to make, and it was used to collect some initial

data. However, the servomotor could not maintain a constant output velocity, and the laser cut rack and pinion were prone to cracking. Additionally, the gear teeth were too coarse to allow the really low flow rates that were of interest based on the first round of data, so a lead screw assembly was the natural option for the second prototype. A more robust apparatus also meant less variation between trials since it has never to date needed any adjustment or repair.

In designing the syringe pump, one of the key parameters is the range in which the mass flow rate can be varied. This is something that neither of the two papers referenced in this thesis has managed to do. This section outlines the important parameters to keep in mind when designing a syringe pump for this purpose.

The mass flow was calculated by dividing the collected fluid mass by the cycle time. In order to evaluate the efficiency of the pump, an analytical expression for the expected flow rate was derived.

$$\dot{V} = \frac{-\pi D_{piston}^2 p s}{2250 \Delta t} \quad (1)$$

where p is the pitch of the lead screw, s is the step size of the stepper motor, and Δt is the pause that the user would use to limit the angular velocity.

Dividing the actual mass flow rate by the ideal mass flow rate gives us the efficiency of the pump and allows us to quickly calculate the number of steps to command the motor to get a given sample size at a given rate. The losses presumably come from backlash in the lead screw assembly as well as expansion effects in the air due to the induced pressure change. If great accuracy is desired, backlash can be eliminated by the simple expedient of moving past the starting point by a distance exceeding the worst-case backlash when the cycle is reversed and then moving into the start position along the same direction that the carriage will move during the collection cycle. The compressibility effects are found to be small for sample columns on the order of 10 cm when making a conservative estimate of the expansion of the air due to the weight of the fluid column.

How close the flow rate is to being uniform depends on the properties of the fluid as well as the geometry of the constriction because air is compressible and therefore acts like a spring. If you lift a weight at constant velocity by pulling on a spring, then there will be a transient region where the spring stretches until it reaches force equilibrium, but then the spring will stay one length as long as you keep moving it at constant velocity. However, if your weight suddenly snagged on something and then came loose your weight would start oscillating. Similarly, if the particles suddenly jam the opening, the will expand to increase the pressure gradient. If the particles then unjam, the flow will speed up to re-establish the force balance. Whether the flow oscillates or not, it will for certain accelerate for a time, meaning that the problem with Haw's experiment having a non-uniform flow rate still exists to some degree. That is, if the particles actually jam and unjam the way that Haw describes. If the particles simply jam early on and stay that way until the piston stops moving, then the greater part of the experiment will still be conducted at a constant flow rate. An interesting modification to my setup might be if the velocity of the rising fluid column could be sampled

continuously. One could then observe a jamming by detecting a sudden deceleration. If this was followed by an acceleration that could indicate that the particles have unjammed again

The flow tubes were cut out of acrylic tubing. On one side a push-valve was stripped of its threads and epoxied in place. For the constriction, a length of copper tube was first epoxied into a laser cut acrylic bushing with an outer diameter that fitted closely inside the flow tube. This bushing was then epoxied into the other side of the flow tube. The epoxy sealed the flow tube well enough that it would not leak when filled completely with water. This was confirmed by testing the tubes with the syringe pump.

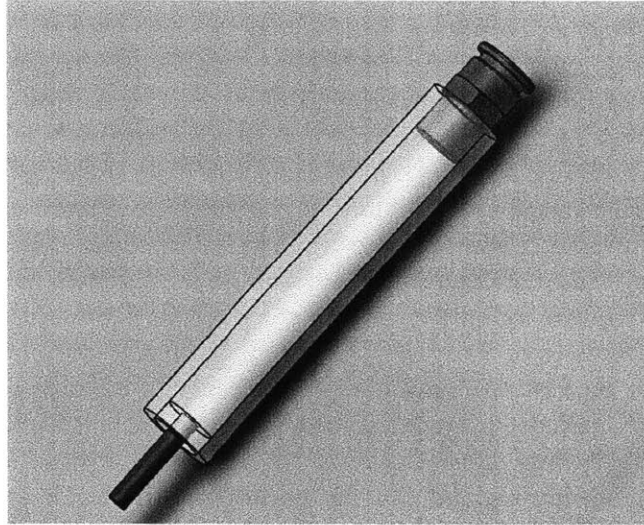


Fig.2 *Solidworks representation of a typical flow tube*

1.4 DATA COLLECTION USING MATLAB

Perhaps the largest challenge in any experiment attempting to measure self-filtration is finding an accurate and repeatable way to measure volume fraction. Haw [1] took perhaps the most direct approach, namely allowing the liquid in each sample to evaporate and then measuring the mass of the remaining solid, effectively measuring the “mass fraction” rather than the volume fraction. Two samples of identical volume fraction were prepared, and then a known volume of suspension was scooped up from one sample while a syringe was used to extract the same volume of suspension from the second sample. He then allowed all liquid to evaporate from each sample and weighed the remaining solid to calculate the difference in mass fraction between the two collected samples. The mass fraction was then converted to volume fraction by assuming known densities of particles and fluid respectively.

One drawback of this approach is that evaporation is a slow process. Furthermore, since we achieve neutral buoyancy in the suspension of polystyrene beads in saltwater solution by regulating the salt content (and consequently the density), this method would require the particles to first be cleaned to remove the salt. We did devise a method for cleaning particles by trapping them in a tube sealed with a fine mesh and running tap water through. However, this is also time consuming, and

complete cleaning of the particles is virtually impossible so there will still be an error. In addition, some particles will be lost during cleaning, which introduces yet another error. This method was nonetheless employed in Kulkarni, although the method for cleaning the particles is not specified.

Another method we considered early on was dyeing a percentage of the particles, and then using an assumption of some probability distribution of dyed particles in the mass of un-dyed particles we could calculate the number of particles using image processing in Matlab. This proved complicated, especially since only particles near the surface of the sample vial could be seen in a photograph.

The method devised for this experiment calculates the required mass fraction for preparing the sample and converts this number to volume fraction via the density of the polystyrene beads. Since the particles in the prepared sample are neutrally buoyant with the fluid phase, the density of the fluid is assumed to be equal to the density of the polystyrene. This density can then be measured in the fluid using a hydrometer. After the particles in the collected sample have settled, a photograph is taken of each sample vial and processed with a Matlab script that measures the column height of the particle sediment and calculates the solid volume of the sediment column using a calibration curve that was found for the internal shape of the sample vials experimentally. The vials were initially approximated as cylinders, but manufacturing variation in the internal geometry from vial to vial produced large uncertainty in the volume measurement when this approximation was used. Instead, known volumes of glass beads were measured in a 10 ml graduated cylinder and then transferred to sample bottles in order to plot a calibration curve for calculating volume based on column height. Glass beads were used because the polystyrene beads have a tendency to stick to glass when dry, introducing uncertainty. The first approach was to use water instead of glass beads for the calibration in order to avoid any variation in the packing of the beads in the graduated cylinder compared to packing in the sample vial. However, the meniscus of the water was so large compared to the height of the water column that there was significant uncertainty in this measurement as well. Instead, the glass beads were measured using a graduated cylinder and then transferred to a sample vial. The assumption is that the beads pack to approximately the same density when poured into the graduated cylinder as when they are poured into the sample vial. Plotting a linear function to the collected data from the glass bead samples yielded the equation

$$V = 5.2719 \times h (\text{cm}^2) - 0.5049 \text{cm}^3 \quad (2)$$

where h is the column height.

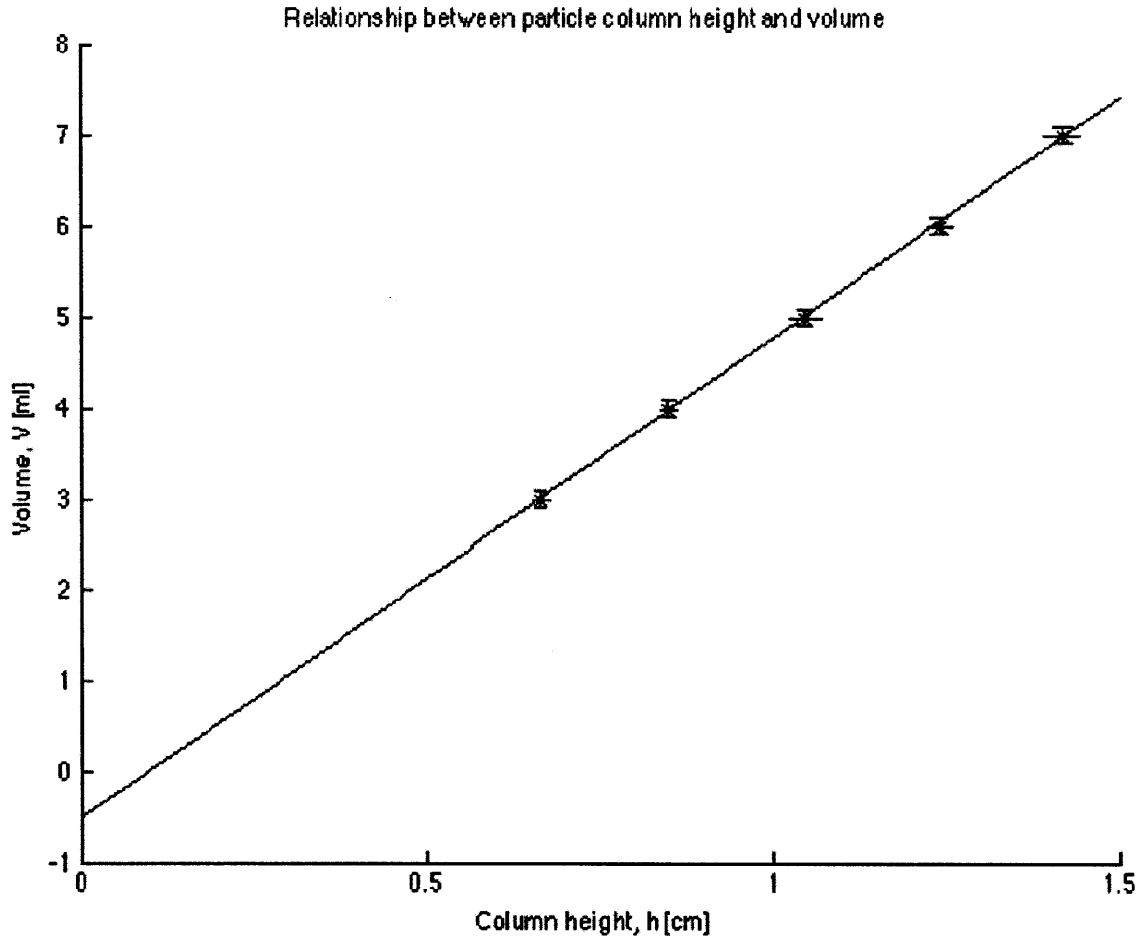


Fig. 3 Linear function fitted to calibration sample data in order to find an equation for volume as a function of particle column height. Vertical error bars indicate instrument uncertainty in the volume measurement due to the graduated cylinder used, while horizontal error bars represent the standard deviations of five separate samples of each measured volume.

In order to find the packing density for polystyrene beads that have settled in tap water, multiple samples of polystyrene beads with known volumes were mixed with tap water and surfactant (the same fluid used to break the neutral buoyancy in collected samples) and photographed after having settled. The packing density, Φ_m , was found by dividing the known volume of particles by the calculated volume of the sediment column. In practice, this was again done by measuring the mass of the calibration samples of particles and converting to volume via density. The mean packing density was calculated to be 0.60 ± 0.0085 .

The script that calculates the volume in a set of samples measures the sediment column height for each sample and plugs this into the volume equation (2). The volume of the column is then multiplied by the constant Φ_m to give the solid volume of polystyrene in the sample. Dividing by the volume collected by the flow tube gives the volume fraction downstream of the constriction.

Chapter 2: Experimental Procedure

2.1 SAMPLE PREPARATION

The external (fluid) phase of the suspension used in all these experiment is prepared by adding table salt to tap water until its density is the same as the density of the polystyrene beads that are used as the internal (particulate) phase, which is specified by the manufacturer as 1.05g/ml. A small amount of Tween 20 surfactant is then added to the fluid so that the beads will not clump together or stick to surfaces after mixing. The fluid phase is made in batches of three liters and kept in a sealed bottle. The particles are mixed with the fluid directly in the beaker from which the flow tube will collect its sample. This is done in order to avoid unknown particle losses that are unavoidable when transferring the suspension from one container to another, causing uncertainty in the initial volume fraction. A beaker is placed on a scale and the required fluid is measured out. Particles are then added until the combined mass reaches the desired value for the sample size used. A stirring rod is used to mix the two phases thoroughly. Care is taken in avoiding excessive bubble formation and the rod is extracted carefully so that few particles stick to it. Although this introduces a small uncertainty, since the mixing procedure is always the same it is a systematic error that is the same for each trial, rather than the unknown error that occurs in pouring out each sample from a common source.

It should be noted, however, that having to mix the suspension for every single trial is by far the slowest step in the sample collection cycle, and future iterations may want to investigate just how large this uncertainty is, because if it is relatively small, then it may be worth pre-mixing suspension in bulk, which would significantly speed up the process. It may be worth noting that Haw transferred suspension from his “bulk sample” (reference sample) by scooping it up, assuming insignificant uncertainty in volume fraction of the scooped suspension compared to the volume fraction of the bulk suspension. If the suspension could be pre-mixed in a large container and then scooped into the beaker, then the experiment could be run significantly faster than with the current procedure.

2.2 DATA COLLECTION

The full experimental procedure following the preparation of the fluid phase was updated repeatedly in response to collected data as well as increased experience with performing the experiment. The final six-step procedure outlined below balances the need for high repeatability with the benefits a higher rate of data collection.

1. The suspension is mixed to the correct volume fraction by calculating the required mass of solid and fluid phase respectively. The solution is added to a beaker on a precision scale until it contains the correct amount. The polystyrene beads are then added until the combined mass corresponds to what was calculated. The suspension is mixed using a stirring rod.
2. An acrylic flow tube with a smaller diameter brass tube at the end is submerged into the suspension vertically from above and held in place with a clamp. (See Fig. 2)
3. A piece of plastic tubing is attached to a pneumatic valve at the opposite end of the flow tube and connected to a programmable syringe pump.
4. The syringe pump induces a negative air pressure gradient in the flow tube at an imposed rate, causing the suspension to be sucked into the flow tube. The new beaker weight is recorded in order to calculate how much suspension has been collected into the flow tube.

5. The collected suspension is expelled into a specimen vial by reversing the pump. Any remaining particles in the tube are flushed out with a mixture of tap water and surfactant into the specimen vial. The vial is then filled up the rest of the way with this lower density flushing fluid, so that the polystyrene beads settle on the bottom over time.
6. After the particles have completely settled, each vial is photographed at close range using a DSLR camera. A Matlab script is used to measure the volume of polystyrene beads in each vial. The volume fraction is then easily calculated as the volume of polystyrene beads divided by the volume of collected suspension that was recorded in step 4.

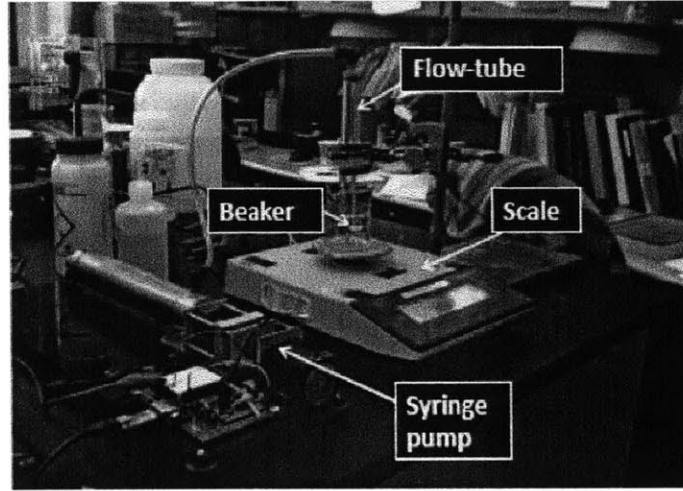


Fig. 4 An annotated overview of the experimental apparatus.

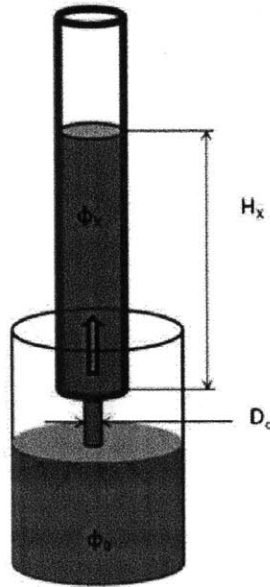


Fig. 5 Conceptual drawing of the flow from the beaker into the flow tube through the constriction.

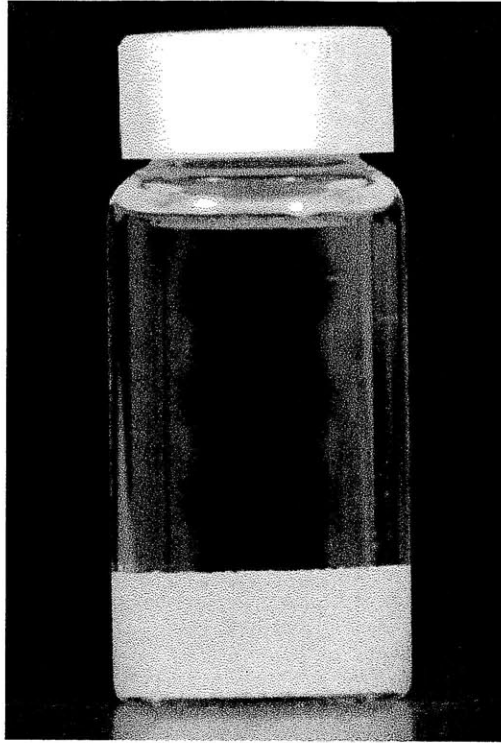


Fig. 6 *Sample vial containing sediment of settled particles.*

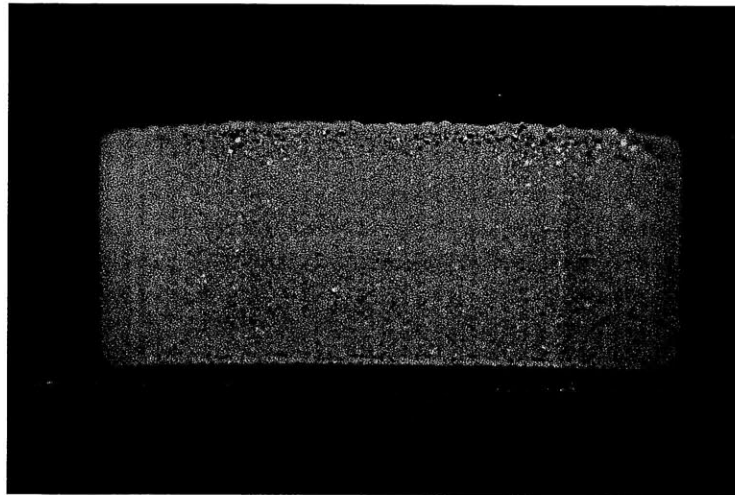


Fig. 7 *Close-up picture with a narrow depth of field improves the repeatability of the Matlab script.*

The syringe pump was designed to allow the user to accurately control the flow rate of suspension, so that the Stokes number

$$Stk = \frac{\rho_p v d^2}{72 \mu D} \quad (2)$$

could be kept constant throughout the pumping cycle. Volume fraction was also plotted against the Reynolds numbers of both the particles as well as the constriction.

$$\text{Re}_d = \frac{\rho v d}{\mu} \quad (2)$$

$$\text{Re}_D = \frac{\rho v D}{\mu_{eff}} \quad (3)$$

For the constriction Reynolds number we cannot use the viscosity of water, and therefore an effective viscosity of the suspension was approximated to zeroth order as being

$$\mu_{eff} = \frac{\mu}{\left(1 - \frac{\phi_0}{\phi_m}\right)^{5/2}} \quad (4)$$

The Stokes number is more relevant for this type of flow than the Reynolds number since the Stokes number expresses how well suspended particles follow the streamlines of the fluid in which they are suspended. In all of the trials presented in this thesis the Stokes number is much smaller than unity (ranging from about 0.0002-0.02), indicating that the particles follow the streamlines of the fluid closely. However, each plot ended up looking qualitatively identical regardless of which of the three dimensionless numbers were used, so the Stokes number was chosen to represent the flow.

The procedure described above was the result of several iterations, where each step was optimized for accuracy, repeatability and speed. Early experiments were conducted using a somewhat different procedure, which is outlined in Appendix B for comparison.

See Appendix A for an error analysis for this first round of trials. Note that this procedure has a calculated relative uncertainty of about 0.96% for initial volume fraction of $\Phi_0=0.51$, which is better than the precision for the most recent procedure. However, it used to take up to 20 minutes in total to get a single data point using this procedure, while the most recent procedure on average takes about 5 minutes. Most of the work this term has been to improve the repeatability of this new and faster procedure, and the data collected has mostly been used to test the repeatability of these experiments. Each step of the new procedure has been carefully tested in order to find any systematic sources of error and minimize them. See chapter 3 for more details on error analysis.

2.3 DATA PROCESSING

Once the sample vials have been photographed, they are stored in a folder labeled with date and trial number. A Matlab script opens each image in order, and the user first calibrates to real world units by clicking the top and bottom of the bottle cap, which has known dimensions. For each sample that follows, the user simply clicks the top and bottom of the particle column, with the first click establishing a vertical guideline to ensure that the column is measured along a single vertical line of pixels. The script outputs a list of volumes corresponding to each sample in the trial, which is placed in a spreadsheet and then used for calculating downstream volume fraction.

3.1 ERROR MODEL

Each step of the experiment was tested for repeatability wherever possible. Otherwise care was taken to minimize experimental error. Following the chain of events outlined in section 2.2, the sources of errors are summarized as:

1. Mixing the polystyrene beads with saltwater has the following sources of error:
 - a. Scale precision: $\pm 0.01\text{g}$
 - b. Density hydrometer $\pm 0.001\text{ g/ml}$
 - c. Loss of particles and saltwater sticking to stirring rod in mixing. (typically reduces mass by 0.03-0.05 % according to observation)
2. Submersion depth did not affect downstream volume fraction but was observed to affect repeatability of the collection process since data points collected from trials with a submersion depth of 0.2 inches were qualitatively more scattered than data points from trials where the submersion depth was 1 inch (see fig. 10). Further study is needed to confirm this, however, due to the large uncertainty in the current experiment.
3. Incomplete seals along the air path occasionally caused backflow as the tube was raised out of the beaker to be deposited into the specimen vial. This was one or two droplets, representing a loss about 0.01-0.02 g of the sample
4. The scale was again used to measure how much suspension remained in the beaker after the pump cycle.
 - a. Scale precision: $\pm 0.01\text{g}$
5. The transfer process from the flow tube to the sample vial was very effective and never resulted in a loss of more than 10 or so particles sticking to the tube wall. This is too small a contribution to affect measurements.
6. In situ measurement of the sediment column has the most significant errors. Errors come from the following sources:
 - a. Variation in packing density
 - b. Uneven surface of particle column
 - c. Variation between photos due to bottle placement and sloping surface of particle column affecting optical perspective.
 - d. Manufacturing variation in bottle internal geometry causing error in volume calculation based on column height.

For every directly measured quantity, the instrument precision was used. For quantities where the precision was unknown, a statistical approach was taken and the precision estimated within 95% confidence bounds. Since sample sizes were small, a t-statistical method was employed. The uncertainty in a quantity that is calculated from other measured quantities (such as volume fraction) was estimated using a propagation of uncertainties method. An example of this is shown in Appendix A for an earlier experimental procedure. The methods for error propagation and

estimating uncertainty using a t-statistical method were adapted from a lecture by Prof. Ian Hunter [4]. The estimated uncertainties for the most recent procedure are summarized in Table 1.

Table 1: *Estimated uncertainties due to various sources of error. Error estimations based on scatter in collected data unless indicated in parentheses.*

Source of Error	Absolute Uncertainty	Mean value of measured quantity	Relative Uncertainty
Mass of Collected Sample (scale)	± 0.01 g	8 g	$\pm 0.13\%$
Volume Calibration	± 0.07 ml	2.86 ml	$\pm 2.45\%$
Packing Density	± 0.0085	0.6	$\pm 1.42\%$
Initial Volume Fraction Measurement (scale, hydrometer)	-	-	$\pm 0.026\%$
Downstream Volume Fraction Measurement	± 0.0061	0.47	$\pm 1.31\%$
Matlab Processing	0.0014	0.41	$\pm 0.35\%$

By far the largest source of error is in the calculation of the volume of the particle sediment column, which has an uncertainty of about 2.45%. This severely impacts the repeatability of the measurements. Note that both packing density and downstream volume fraction have errors that have been propagated from that of the volume calibration uncertainty. The internal geometry of the vials is too irregular to be approximated as a cylinder, and the bottles vary with respect to one another. In addition, the inner diameter of a sample vial cannot be measured with available tools (calipers) without cutting off the bottleneck of the vial, which would destroy it. If we had access to rectangular cuvettes, such as were used in Haw's experiment, the internal dimensions of each sample vial could be measured using calipers, which would reduce the uncertainty in volume calculation to about 0.1% if we assume column height is on the order of 1 inch and the calipers have a 0.001 inch precision. The total uncertainty of downstream volume fraction would then drop to an estimated 0.17%, which might enable more meaningful quantitative data to be collected.

The uncertainty associated with processing a typical image in Matlab was estimated by processing the same set of 15 pictures 5 times. The mean standard deviation of calculated downstream volume fraction obtained from the 5 measurements in each of the 15 pictures is 0.0012. Using the t-statistical method gives an average absolute uncertainty of 0.0014, or an average relative uncertainty of 0.35% when divided by the average downstream volume fraction.

3.2 DESCRIPTION OF FINDINGS

Although this thesis has been primarily concerned with developing a repeatable and useful experiment for measuring self-filtration, a large amount of data was collected in the process. The reader should note that the accuracy and repeatability are not very good for many of these data sets, because the procedure as well as the actual hardware and software were being updated regularly. However, qualitative trends worth mentioning will be discussed briefly in this section, and the plotted data shown.

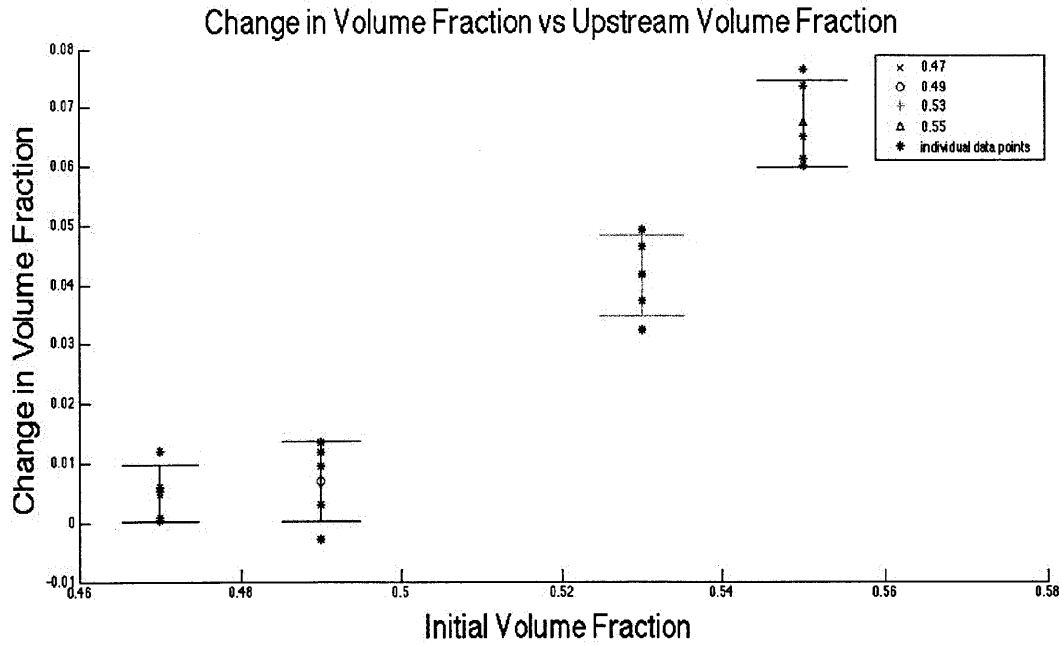


Fig. 8 Reduction in volume fraction vs. initial volume fraction after the suspension has passed through a constriction at $Stk=0.0213$. The mean values for the four initial volume fractions used are plotted with error bars, with the individual data points superimposed as black asterisks.

Fig. 6 shows clearly that larger initial volume fraction results in a larger change in volume fraction as the suspension of PB-4 beads (average diameter $d=0.008$ in) passes through a constriction of diameter $D=5/32$ in. This result agrees qualitatively with Haw's observations. We can see that trials with initial volume fraction of 0.47 and 0.49 seem to be below or close to the limiting value where self-filtration starts to occur as described by Haw, but we can see significant self-filtration (around 8% reduction in volume fraction) occur when the initial volume fraction is 0.53, and even more pronounced when the initial volume fraction is 0.55 (about 13%).

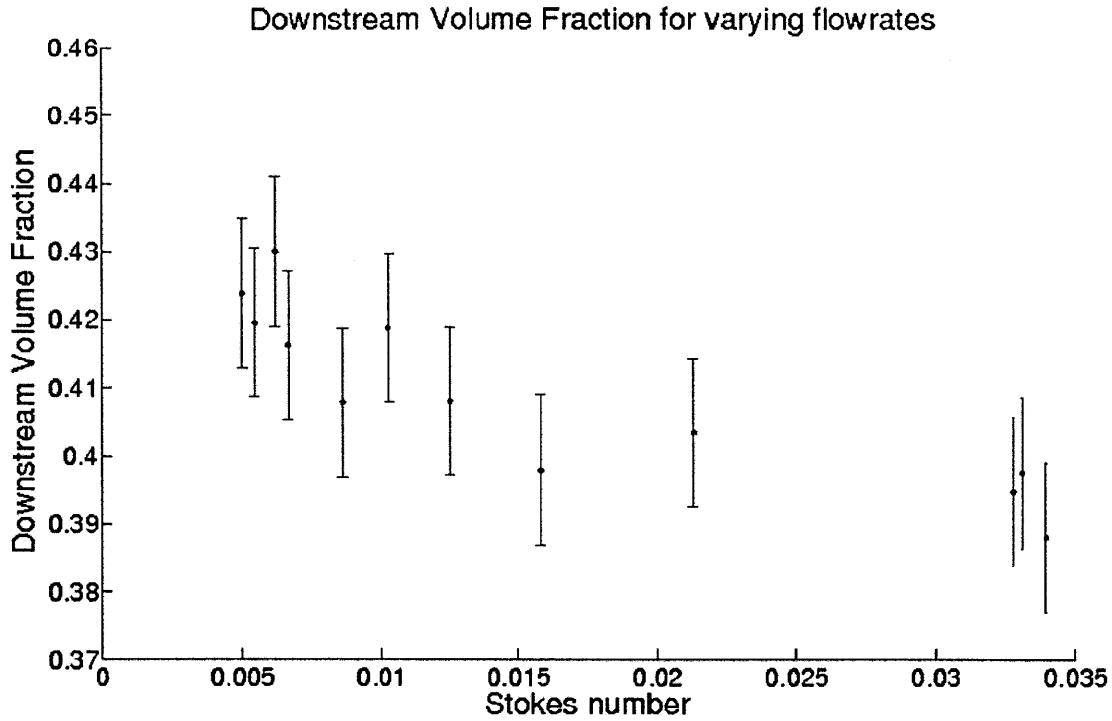


Fig. 9 Downstream volume fraction decreases as flow rate is increased (and thus the Stokes Number is also increased) indicating that high flow rates cause more significant self-filtration than low flow rates. Each data point is plotted with error bars corresponding to estimated 95% confidence uncertainty.

The data set plotted in fig. 7 used PB-2.5 beads (average diameter $d=0.017$ in) in a suspension with a volume fraction of 0.47 passing through the same constriction as in fig. 6 ($D=5/32$ in). In this case, there is significant self-filtration at $\Phi_0=0.47$, indicating that the average bead diameter is an important factor affecting self-filtration.

The question then naturally arises of whether it is really the ratio of particle diameter to constriction diameter that affects self-filtration? Using a larger constriction diameter of 0.302 inches and the larger particle size PB-1 (average diameter $d=0.0315$ in), we get approximately the same ratio d/D as for the data plotted in fig. 7 and we can plot it for a similar range of Stokes numbers.

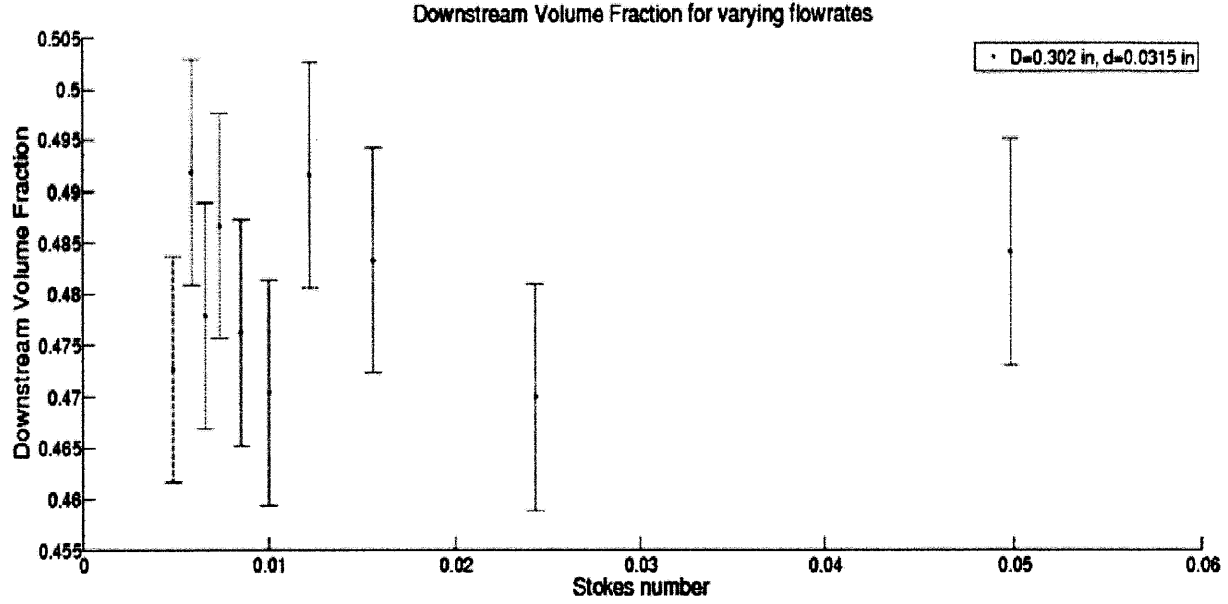


Fig. 10 *Scaling both particle and constriction diameter together surprisingly appears to give no correlation between flow rate and downstream volume fraction.*

This data set is one of those taken before the most recent procedure was developed, and so the actual numbers are inaccurate. This set had an initial volume fraction of 0.47, so downstream volume fraction was apparently over-estimated for this set. However, qualitatively we can still see that there is no visible correlation in this data, so it does not appear to be so as simple as that scaling constriction and particle diameter together gives the same self-filtration behavior.

A final remark should be made about the effect of submersion depth of the constriction tube into beaker of prepared suspension that was mentioned in section 3.1. While submersion depth does not appear to affect self-filtration in a systematic way, there does appear to be increased uncertainty associated with a shallower submersion depth. What does seem to make a difference is the amount of suspension that the sample is collected from. For samples where about 8 ml of fluid is drawn from an initial volume of 20 ml suspension we observe more self-filtration compared with samples where 8 ml of suspension is drawn from an initial volume of 100 ml suspension. Figure 11 summarizes the absolute uncertainty in Volume Fraction for different combinations of submersion depth and initial upstream suspension volume.

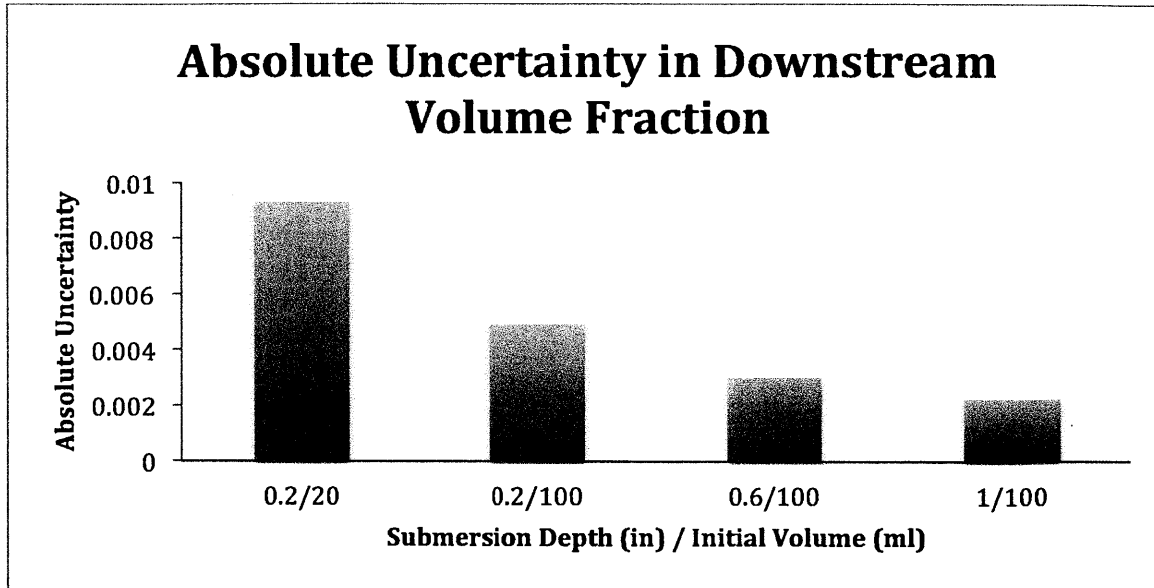


Fig. 11 Absolute Uncertainty in downstream volume fraction for combinations of submersion depth and initial volume of suspension in the prepared sample. We observe that deeper submersion combined with a large initial sample gives the most repeatable results.

For the same set of data, we also observe that the average downstream volume fraction for samples with an initial volume of 100 ml is $\Phi_x = 0.42$ while the average downstream volume fraction for samples with an initial volume of 20 ml is $\Phi_x = 0.40$. In other words, more self-filtration occurs when the initial volume is small. The drop in self-filtration (increase in downstream volume fraction) seen by increasing initial volume makes intuitive sense because as more and more fluid is filtered out of the upstream part of the sample, then the upstream volume fraction increases at the same time that downstream volume fraction decreases. We saw from fig. 6 and [1] that increased upstream volume fraction increases self-filtration effects, so when 40% of the initial volume is extracted it makes sense that the opposite effect of self-filtration takes place upstream in order for mass to be conserved. By increasing the initial volume upstream to 100 ml, the particles that are left behind by self-filtration no longer have a great impact on increasing the upstream volume fraction and less self-filtration is observed.

3.3 SUMMARY AND FUTURE WORK

An experiment has been designed and tested for measuring Self-Filtration effects in non-colloidal suspensions. The repeatability was not good enough for quantitative analysis in any meaningful sense, but a solution has been proposed in changing from generic quasi-cylindrical sample vials to an easier to measure geometry such as rectangular cuvettes. The method of propagated uncertainty then predicts an uncertainty of only 0.17 % for the downstream volume fraction measurement. Other future work that can be undertaken would be to run a new set of trials with the improved repeatability in order to quantify the phenomenon better and perhaps fit functions to the data that could be used in combination with theory to predict the flow of suspensions through a constriction.

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APPENDIX A

Error Analysis for $\phi = 0.51$

Parameter	Symbol	Numerical Value	Source
Water mass	m_w	$m_w := 12.87\text{gm}$	Weighed on scale
Precision of m_w	u_{mw}	$u_{mw} := 0.01\text{gm}$	Specs from Sartorius
Particles suspended	m_p	$m_p := 13.40\text{gm}$	Weighed on scale
Precision of m_p	u_{mp}	$u_{mp} := 0.01\text{gm}$	Specs from Sartorius
Volume collected	V_x	$V_x := \pi \left(\frac{1}{4} \text{in} \right)^2 \cdot 4.75 \text{in} + \pi \left(\frac{5}{64} \text{in} \right)^2 \cdot 1.1 \text{in}$ $V_x = 15.629 \cdot \text{mL}$	Measured with ruler then calipers
Precision of V_x	u_{Vx}	$u_{Vx} := (0.5\text{mm} + .001\text{in}) \cdot \left[\pi \cdot \left(\frac{1}{4} \text{in} \right)^2 + \pi \cdot \left(\frac{5}{64} \text{in} \right)^2 \right]$ $u_{Vx} = 0.073 \cdot \text{mL}$	
Packed Volume of particles	V_p	$V_p := 8.5\text{mL}$	Measured with graduated cylinder
Precision of V_p	u_{Vp}	$u_{Vp} := 0.05\text{mL}$	0.1 ml increments

Density of neutral fluid (same as particle density)

ρ

$$\rho := 1051 \frac{\text{kg}}{\text{m}^3}$$

Measured with hydrometer

Specs from VWR

Precision of ρ

u_ρ

$$u_\rho := 1 \frac{\text{kg}}{\text{m}^3}$$

Measured volume of particles in calibration trials

V_m

$$V_m := 90 \text{ mL}$$

$$u_{V_m} := 0.5 \text{ mL}$$

Measured with graduated cylinder and then calculated

Propagation of Error

Water volume

V_w

$$V_w := \frac{m_w}{\rho} = 0.012 \text{ L}$$

Precision of V_w

u_{V_w}

$$u_{V_w} := \sqrt{\left(\frac{\partial}{\partial m_w} \frac{m_w}{\rho} \cdot u_{m_w}\right)^2 + \left(\frac{\partial}{\partial \rho} \frac{m_w}{\rho} \cdot u_\rho\right)^2}$$

$$u_{V_w} = 0.015 \text{ mL}$$

Experimentally determined packing fraction

ϕ_m

$$\phi_m := .613$$

average error

Precision of ϕ_m

u_{ϕ_m}

$$u_{\phi_m} := .71\%$$

Volume fraction for suspension mixing

$$\phi_0 := \frac{m_p}{m_p + V_w \cdot \rho} = 0.51$$

Precision in
volume fraction
for suspension
mixing

$$u_{\phi 0} = \sqrt{\left(\frac{\partial}{\partial m_p} \frac{m_p}{m_p + 4.5V_w \cdot \rho} \cdot u_{mp}\right)^2 + \left(\frac{\partial}{\partial V_w} \frac{m_p}{m_p + 4.5V_w \cdot \rho} \cdot u_{Vw}\right)^2 + \left(\frac{\partial}{\partial \rho} \frac{m_p}{m_p + 4.5V_w \cdot \rho} \cdot u_{\rho}\right)^2}$$

$$u_{\phi 0} = 0.026\%$$

Volume fraction
for collected
fluid

$$\phi_x := \frac{V_p \cdot \phi_m}{V_x} = 0.333$$

Precision in
volume fraction
for collected
fluid

$$u_{\phi x} = \sqrt{\left(\frac{\partial}{\partial V_p} \frac{V_p \cdot \phi_m}{V_x} \cdot u_{Vp}\right)^2 + \left(\frac{\partial}{\partial V_x} \frac{V_p \cdot \phi_m}{V_x} \cdot u_{Vx}\right)^2 + \left(\frac{\partial}{\partial \phi_m} \frac{V_p \cdot \phi_m}{V_x} \cdot u_{\phi m}\right)^2}$$

$$u_{\phi x} = 0.46\%$$

Volume fraction

ϕ_0

$$\phi_0 = 0.51$$

**Overall uncertainty in
volume fraction for entire
experiment**

u_{ϕ}

$$u_{\phi} := u_{\phi x} + u_{\phi 0} = 4.866 \times 10^{-3}$$

$$\phi = 0.51 \pm 0.0049$$

APPENDIX B

Outline of earlier experimental procedure. Steps 1-3 were the same as the most recent procedure, and are therefore omitted.

4. The column height of the collected fluid was measured with calipers from a reference point. This reference point was drawn on the outside of the flow tube with a pen at the column height my analytical model predicted that the collected fluid column would reach. The calipers were used to measure the amount by which the column height of the collected sample deviated from this reference point. The idea was that this gave better repeatability than measuring the whole length of the column height each time, and it made measurements faster. The collected volume was then calculated based on the inner diameter of the acrylic tube and the measured column height. The volume of fluid that remained in the beaker after collection (the “bulk sample”) was also measured, but using a 100 ml graduated cylinder.
5. The collected fluid was expelled using a similar technique as in the most recent procedure, but the sample was collected in a 10 ml graduated cylinder, which was then filled up with tap water in order to make the particles settle. Rather than waiting for this to happen over time, however, the graduated cylinder was repeatedly tapped against the table top until no further decrease in column height was measurable with the resolution of the graduated cylinder, and the volume of the sediment column was read off the graduated cylinder and recorded. The same procedure was conducted on the bulk sample by filling up the larger graduated cylinder.
6. Since both the collected sample and bulk sample particle and suspension volumes were measured, it was possible to calculate the volume of particles in the collected sample as well as the packing density of the particles for each sampling cycle. The packing density was recorded so that for later trials it would not be necessary to collect and measure two samples each time, but rather use the average packing density to calculate the volume of the collected sample directly, which then cuts the time it takes to produce a single data point by almost 50%